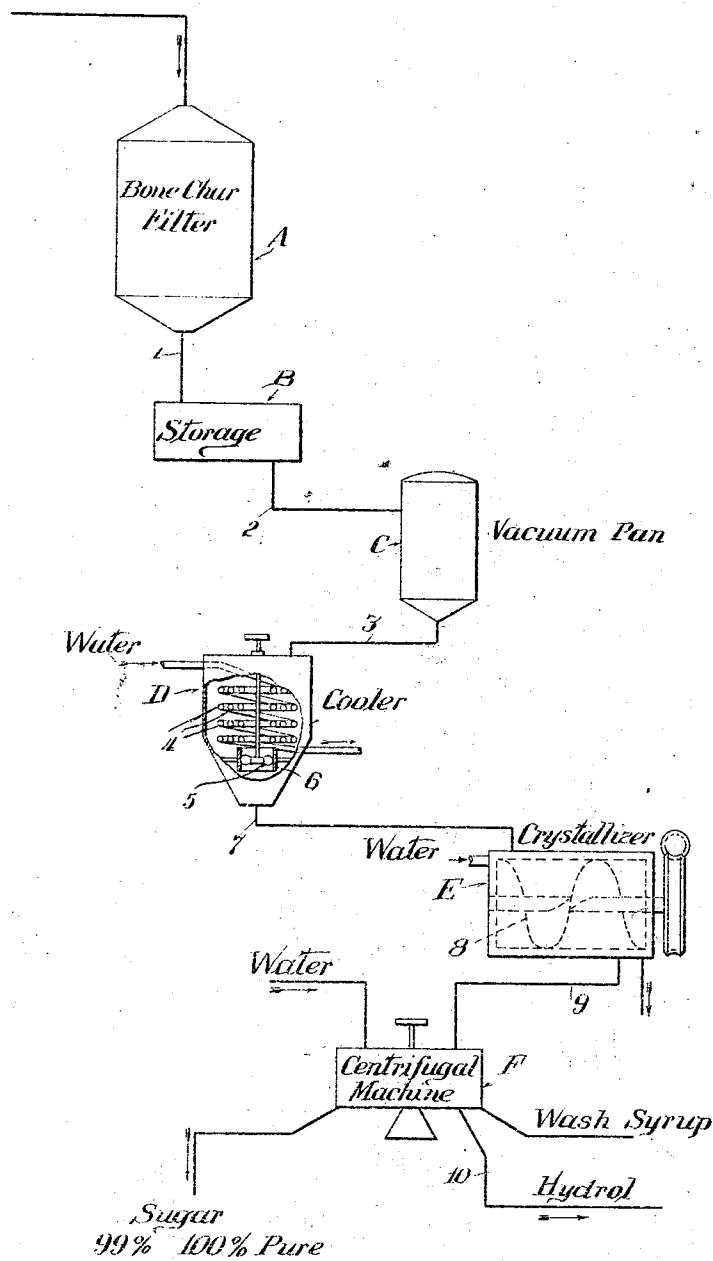


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W. B. NEWKIRK
MANUFACTURE OF DEXTROSE
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UNITED STATES PATENT OFFICE.

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MANUFACTURE OF DEXTROSE.

Application filed September 10, 1924. Serial No. 736,945.

To all whom it may concern:

Be it known that I, WILLIAM B. NEWKIRK, a citizen of the United States, residing at Riverside, in the county of Cook and State of Illinois, have invented certain new and useful Improvements in the Manufacture of Dextrose, of which the following is a specification.

My invention relates to certain improvements upon the method of making crystalline dextrose disclosed in my Patent No. 1,471,347 granted October 23, 1923; these improvements consisting primarily, first, in the employment of a relatively large amount of dextrose crystals as seed; second, in the use, as seed for each successive batch, of a mixture of crystals and mother liquor before the same has had time to set or harden, termed "foots" herein which is left, for the purpose, in the crystallizer when the preceding batch is removed therefrom to the centrifugals; and third, (this feature being optional) in cooling the concentrated solution before it is introduced into the crystallizer to such a temperature that when mixed with the foots from the last operation it will be at just the proper temperature for the induction of new crystals or the building up of the crystals introduced initially as seed.

The process may be employed advantageously for the manufacture either of hydrate or anhydrous dextrose. In either case the success of the operation depends upon obtaining a mass of crystals of normal crystal form for the type being manufactured, that is, substantial prisms of appreciable cross sectional dimensions both ways with respect to the length of the crystal instead of the flake-like or needle-like, or very minute crystals, which are collectively termed "false grain." With the crystals of the normal form and size the magma may be purged of its mother liquor completely, or substantially so, by centrifuging. If the magma contains any very considerable quantity of false grain complete purging becomes impossible because of the filling of voids by the minute crystals or broken particles of the flakes and needles.

To obtain a complete purgable magma (and hence a substantially pure sugar) from a converted liquor of ordinary purity

(89%-90% dextrose on the basis of dry substances) the following conditions appear to be essential: (1) The solid phase dextrose must be uniformly distributed through the solution, this being accomplished by keeping magma in motion by means of an agitator; (2) the seed crystals used for inducing crystallization of the dextrose in solution should be of the same crystal type (either hydrate or anhydrous) as the crystals to be induced; (3) crystallization should take place at relatively high temperatures (within, generally speaking, the anhydrous range) for the anhydride, and at lower temperatures (the hydrate range) for the hydrate; (4) the degree of supersaturation should be in proportion to the other factors. If supersaturation is too great, either in the magma as a whole or locally, spontaneous crystallization takes place, with the formation of false grain, instead of the natural induction and growth of crystals of normal size and form. On the other hand, if the degree of supersaturation is too small the time required is unduly lengthened and the operation made expensive. The degree of supersaturation is dependent upon the density of the solution, the temperature drop below the saturation temperature, and the amount and character of the impurities in the solution.

These are the general conditions for successful operation as set forth in my former patent which discloses a practical and workable method of procedure based upon such conditions for making a high purity product. The patented method, however, involves considerable care and attention in the way in which the operations are carried out in order that uniformly satisfactory results should be obtained. In manufacture on a large scale such rigorous care and attention to details are difficult to maintain, and as a consequence unpurgable magmas are likely to be turned out from time to time due to carelessness or to variations in the character of the converted liquors used.

Seeking to remedy these difficulties I have since discovered that the control of the process is greatly facilitated and uniform purity of output better assured by using a large quantity of seed in the manner indicated. That is to say, I use seed in such an amount

that the quantity of the solid phase in the solution is a significant factor of control. It may, in fact, be the dominant or controlling factor so that the other factors, those of density, temperature and purity, become of secondary importance. Experience has proven that with a large amount of the solid phase of the right crystal type evenly distributed throughout the solution and so maintained from beginning to end of the crystallization period, supersaturation may be safely maintained at a higher degree than has been heretofore feasible; a wider range of crystallizing temperatures is made feasible with consequent possibility of increase in yield; variations in temperature and in the purity and the density of the solution are of less importance as disturbing factors; the period of crystallization may be shortened; and when necessary to obtain an especially large grained product, which the trade may demand, it is possible to produce such product by using a liquor at a density calculated at its temperature when mixed with the seed to bring about only the growth of the seed crystals introduced without causing induction to any large extent, of new crystals.

By employing instead of dry seed foots from a previous batch there is introduced into the new solution both solid phase and liquid phase in nascent condition, that is, in a condition of maximum activity. Moreover, the process becomes continuous so that having once built up (possibly after the production of several batches of more or less impure crystals) a magma of satisfactory character for purging, the foots left in the crystallizer from the satisfactory batch, and from each subsequent batch, are determinative, in large measure, of the character of the subsequently made batches.

By the artificial and relatively rapid cooling of the concentrated solution before it is brought into contact with the seed (this being a preferred feature of the improved process), the induction of crystals may be initiated with more certainty at the proper temperature for obtaining the right type, size and form of the crystals induced. Preferably the pre-cooling of the liquor is carried to a substantial supersaturation of the solution but not far enough to induce crystallization to any appreciable degree. This is possible because of the large supersaturation which dextrose solutions can carry. The addition of relatively large quantities of dextrose crystals at low temperature initiates crystallization by a further reduction of the temperature of the solution and by the presence of additional dextrose in the solid phase. The fact that it is possible to reduce the temperature very considerably during crystallization, because of the large quantity of the solid phase present, makes

feasible the use in the new batch of foots from a previous batch at such low temperature as to materially cool the new batch.

The annexed drawing is a flow sheet diagram illustrating the successive steps of the process as it is preferably practiced.

The preferred method of manufacturing the hydrate may be described as follows:

The starch is converted, preferably, by the usual method of acid hydrolysis, to as high a dextrose content as practical, 89% to 90% for example, based on dry substances. The converted liquor is then preferably filtered by being run through a bone char filter A (referring to the annexed drawing). The filtered liquor is then run through a pipe 1 into a storage tank B from which it passes by pipe 2 to a vacuum pan C. The liquor is concentrated in the vacuum pan to a density of from approximately 38° Baumé to 45° Baumé. The preferable density is about 40° Baumé. The concentration in the vacuum pan may be conducted, according to circumstances, at any temperature from 130° to 180° Fahrenheit. From the vacuum pan the liquor is introduced by pipe 3 into a cooler D which is preferably provided with a number of cold water coils 4 and with a propeller 5 arranged in a sleeve 6 near the bottom of the cooler. When the liquor has been sufficiently cooled it is run through pipe 7 to the crystallizer E which is preferably water jacketed and provided with a revoluble agitator 8. Here it is mixed with crystallized dextrose, preferably in the form of foots left from the manufacture of the last batch. One of the advantages of foots over dry seed is that the foots can be more easily dispersed throughout the new batch of liquor than the dry seed. Another is that the crystals are in a growing state. Moreover, the mother liquor in the foots contain some dextrose in a state ready to crystallize and this influences the crystallization of the new liquor. The mother liquor, therefore, as well as the crystals in the foots, is a factor influencing crystallization of the new solution, and some advantage, though possibly small, would accrue from introducing mother liquor alone into the new batch. Preferably the amount of the "foot" will be approximately 40% of the new batch (assuming a purity of the converted liquor of about 89%), the other 60% of the batch consisting of fresh converted and concentrated liquor from cooler D. These proportions may be varied over quite a wide range. The amount of the foots may, for example, vary from 30% to 45% of the batch with a liquor of approximately the purity mentioned. The solid phase will ordinarily be from four-tenths to six-tenths of the foots, the remainder being its mother liquor. The proportions of solid phase to liquid may therefore vary approximately from 10 to 40 parts of solids to 100 parts

liquid. The fresh liquor is preferably run into the crystallizer before the foots therein have had a chance to completely cool and set, for the reasons stated, and the temperature to which the concentrated liquor is cooled is calculated so that when this liquor is mixed with the foots, which is likely to be at a considerably lower temperature than the liquor, the mixture will have the temperature desired for initiating the crystallizing operation. This temperature is preferably 105° Fahrenheit, or thereabouts. It may be considerably higher than this, for example, as high as 130° Fahrenheit, depending upon the character of the converted liquor particularly as to impurities. While as high a temperature as 130° is within the anhydrous range, as ordinarily considered, that is, the range of temperatures favorable, generally speaking, to the production of anhydrous crystals, the predominance of the large quantity of the hydrate solid phase prevents the formation either of anhydrous crystals or false grain. The temperature of the magma in the crystallizer is preferably maintained for a time at approximately 105° Fahrenheit. If the heat developed by the crystallization is in excess of radiation the mass may be cooled by circulating water through the jacket of the crystallizer. After the crystallization of the converted liquor has gotten well started the mass may be cooled down by the circulation of water through the jacket until, at the end of the operation, quite a low temperature is reached, for example, as low as 80° Fahrenheit or even lower. The condition of the material will determine the extent to which the temperature can be reduced, the aim being to obtain as great a temperature drop below saturation as possible, to obtain a maximum yield, without detrimentally affecting the character of the crystals.

The improved results, in these respects, are due to the large proportion of the foots introduced into each batch at the start. While the ratio of 40% foots to 60% fresh liquor is the optimum proportion as I regard it, having in view normal runs of liquors, it will be possible, in fact desirable in some cases, to vary this ratio both ways within the limits of 30% foots to 70% liquor and 45% foots to 55% liquor, as above referred to, or even further. With the ratios given the amount of the seed used (roughly 10 to 40 parts the solid phase to 100 parts of the liquid) is very much greater than has been heretofore customary so far as I am aware. Usually a very small amount of seed, hardly more than one or two per cent and frequently less, has been used or advocated. The important consideration is that enough seed of the right type and preferably in the growing or developing form (foots) should be used so that the quantity of the solid phase is

a significant factor in the control of crystallization during both the periods of induction and growth. Heretofore in the manufacture of dextrose or grape sugar the amounts of seed used have been so small that the quantity of solid phase introduced has had no significance, from a practical point of view, in influencing crystallization.

When the crystallizing operation is completed 60% (more or less) of the magma is run through pipe 9 into the centrifugal machine F, the remainder of the batch being left in the crystallizer and mixed as soon as possible with a new batch of concentrated and cooled converted liquor. The hydrol or mother liquor passes out of the centrifugal machine through pipe 10 and may be subjected to a second and, if preferred, to a third crystallizing operation as described in my former patent, the re-treatments of the liquor, however, preferably following the improved method hereinbefore described but with certain modifications to be indicated.

As in the process of my former patent, my present process involves washing the sugar in the centrifugal machine F by means of a water jet introduced into the machine through pipe 11 after the hydrol has been extracted. 12 designates a pipe for carrying off the wash water which wash water may be used subsequently in the process as described in my former patent. The sugar after being washed is scraped from the basket of the centrifugal machine and dried in any suitable manner. With this process it is possible to obtain a sugar having an impurity content of less than one-half of one per cent, and to accomplish this without excessive washing. Sugars having a purity of 99.7% to 99.8% have been made in this way. If the crystallized magma is of such character, due to the false grain present, that it can be centrifuged only with difficulty an excessive amount of washing must be done in order to give a reasonably pure sugar and this reduces batch yield because of the sugar dissolved and carried away by the wash water, thereby adding to the cost of the process.

With reference to the treatment of the hydrol or mother liquor to obtain a second or third yield of dextrose or other dextrose solution for that matter, it may be said that, when hydrate sugar is to be made, the greater the impurities present the smaller will be the amount of seed necessary in order to be sure of obtaining satisfactory results. On the other hand, if anhydrous sugar is desired the smaller the amount of impurities present the smaller will be the amount of seed required. With a pure liquor a suitable anhydrous sugar can be produced without seed. The process of making anhydrous dextrose from pure solutions without seed is not claimed herein specifically as it is the subject of my copending application Serial

No. 730,374, filed August 6, 1924. Consequently in treating first hydrols for manufacture of the hydrate it is possible to obtain the advantages of my present invention, namely, the use of solid phase in sufficient amount to be a significant factor in controlling crystallization, by using foots ranging in quantity from 35% down to 20% of the mixture. For treatment of second hydrols, in which the purity is likely to be still lower the foots may be still more reduced. Generally speaking, the amount of solid phase in the solution should be proportioned to the amount of supersaturation and the velocity of crystallization natural for the solution employed. If the velocity of crystallization is high a large amount of solid phase is required; if the velocity is low a smaller amount; if the supersaturation is great a large amount; if the supersaturation is low a small amount. If supersaturation is high and velocity of crystallization great, the quantity of solid phase must be at a maximum; and if the supersaturation is low and the velocity low the amount of solid phase may be considerably less. Generally speaking, the natural velocity of crystallization is reduced by the presence of impurities, the nature as well as the quantity of the impurities being a factor. Ordinarily with pure solutions, the natural velocity of crystallization will be at a maximum. It will be decreased as the impurity content increases; but as stated, the character of the impurities will alter this relation to some extent, a matter which can be determined empirically in any given case.

The case of the manufacture of the anhydride from pure solutions is an apparent but not a real exception to this rule. The natural velocity of crystallization or tendency to crystallize is, under these conditions, high because of the lack of impurities in the solution; but this is offset by the fact that at the relatively high temperatures used for producing anhydrous crystals the degree of supersaturation is small so that the actual rate of crystallization is slow and, as stated, seed need not be used in order to obtain a purgable magma, or the amount used may be quite small.

By using the proper amount of solid phase it is even possible to dispense with the uniform dispersion of the solid phase on the liquor, particularly if a completely purgable magma, that is, the highest purity sugar is not required.

To sum up: A dextrose solution tends to crystallize in a variety of different ways in accordance with variations in the density, purity and temperature of the solution. Conditions must be controlled so as to bring about crystallization in one of these ways alone, i. e., in a way which will produce crystals of what have been termed of normal,

that is, purgable form and size. The crystal habit of dextrose in either range is toward a form of crystal which is not purgable. My present invention is based upon the discovery that this control of the process may be accomplished, with very great advantage from the view point of economical operation, by introducing into the solution such a quantity of the solid phase that any tendency away from the production of the kind of crystals wanted is counteracted by the influence of the solid phase present. This mode of procedure permits the other conditions ordinarily influencing crystallization to be changed unfavorably if need be, to the formation of normal crystals, that is, crystals of the kind wanted, which may happen either through accident, for example, through accidental variations in the converted liquors used; or intentionally, with a view to economy of operation, i. e., shortening time of crystallization or increasing yield. That is what I mean by saying that my invention contemplates having present the solid phase of the type of product wanted and of normal crystalline form in amount sufficient so that the quantity of the solid phase is a significant factor in controlling crystallization. That is by making the quantity of the solid phase a "significant factor" in control, I mean the presence in the magma of such quantity of the solid phase as will cause to be produced a body of crystals like such controlling solid phase in type and form. The solid phase controls crystallization by overcoming the natural crystal habit which under most conditions is unfavorable to the form of crystals wanted.

A large grained sugar may be obtained in accordance with the process above outlined, by using a converted liquor of relatively low density, say from 38° to 40° Baumé. The initial and final temperatures of the crystallizing stage may be the same as above indicated. The use of a relatively large quantity of foots has a direct bearing upon the production of a large grained sugar. That is, the low gravity of the solution involves, at normal temperatures, so slight a supersaturation that, with a large quantity of the solid phase present in dispersed condition throughout the solution, no new crystals, or at least very few, are induced, and all, or practically all of the available sugar crystallizing out of solution goes to build up the size of the original crystals introduced as seed. To make this operation possible there must, as stated, be present at the start a relatively large number of crystals uniformly dispersed through the magma. The method of making large grained sugar, as described above, is not claimed specifically herein, as it is the subject matter of a divisional application filed October 23, 1924, Ser. No. 745,375.

For the manufacture of anhydrous sugar the same conditions are maintained except that the initial temperature of crystallization will be approximately from 130° Fahrenheit to 145° Fahrenheit for a converted liquor of ordinary purity and the final temperature approximately from 100° Fahrenheit to 120° Fahrenheit. This low final temperature is possible at the end of the operation without formation of hydrate crystals or false grain because of the control of crystallization through the use of relatively large quantities of the anhydrous solid phase. If the hydrol from the first centrifuging operation is re-crystallized it is preferable to perform this operation under conditions which will induce crystallization in the hydrate rather than the anhydrous form as difficulties are experienced in the treatment of hydrols in maintaining the conditions necessary for the production of the anhydride because of the relatively larger quantity of impurities contained in the solution. However, crystallization in the anhydrous form is possible from hydrols, if the hydrol purity is not too low, by using especially large quantities of the solid phase. This is but another application of the general principle involved.

The actual temperatures and densites employed will, in any case, depend, necessarily, upon the grade of sugar to be produced, that is, upon the size of the sugar granules desired in the finished product, and upon the character of the converted liquor in respect to the quantity and nature of its impurities. The duration of the crystallizing operation will also vary considerably, depending upon the character of the material treated. The proportion as between the foots and the liquor for a given batch will also depend upon similar varying conditions. If the proportion of foots to liquor is too great the amount of material handled in any given equipment may be so small as to make the enterprise unprofitable. On the other hand, if the proportion of the foots is too small the crystallization will be slow and the chance of false grain being produced will be enhanced. In matters of detail something must be left to the discretion of the skilled operator. I have endeavored to lay down the general principles governing the improved practices herein described and claimed, and I have stated temperatures, densities and proportions within the limits which I have found practical and desirable in order to obtain the best results from the point of view of high purity of the product and economy of manufacture. The best results can be obtained only by such variations in the described method as may be necessary in order to meet any given set of conditions. In the light of the above disclosure of the principles and preferred practice of my in-

vention these variations will be within the power of the ordinary skilled worker in this art. By the term "single crystal type" used as descriptive of the dextrose crystals I mean crystals either of the hydrate type or the anhydrous type to the substantial exclusion, so far as any detrimental effect is concerned, of crystals of the other type. The term "starch converted dextrose solution" is intended to include not only the liquors direct from the converters but also mother liquors from the centrifugals, or remelts, or mixtures of any of these liquors in any proportions. By the terms "hydrate range" and "anhydrous range" as used without qualifications in the claims I intend the ranges of temperatures favorable to the induction of hydrate and anhydrous products respectively but as such ranges are widened through use of the relatively large quantity of the solid phase as set forth herein.

This application is a continuation in part of my co-pending application Serial No. 705,780 filed April 11, 1924.

I claim:

1. Method of making dextrose from a starch converted dextrose solution which consists in concentrating the solution by heating and subjecting the same to a cooling operation comprising mixing with the solution solid phase dextrose in a quantity and at a temperature sufficient to substantially reduce the temperature of the solution to a point to induce crystallization.
2. Improvement in the method of making dextrose from a starch converted dextrose solution, which consists in maintaining, in substantially uniform dispersion through the solution and from beginning to end of the crystallizing operation, solid phase dextrose in such relatively large amount that the quantity of the solid phase is a significant factor in controlling crystallization.
3. Method of making dextrose from a starch converted dextrose solution which consists in concentrating the solution by heating, cooling the solution to supersaturation without substantial induction of crystals and adding to the solution solid phase dextrose in a quantity and at a temperature to initiate crystallization.
4. Improvement in the method of making dextrose from a starch converted dextrose solution which consists in maintaining, in substantially uniform dispersion through the solution and from beginning to end of the crystallizing operation, solid phase dextrose of substantially a single crystal type and normal crystalline form in such relatively large amount that the quantity of the solid phase is a significant factor in controlling crystallization.
5. Improvement in the method of making dextrose from a starch converted dextrose solution having a purity of approximately

89% to 90% which consists in maintaining in substantially uniform dispersion through the solution and from beginning to end of the crystallizing operation solid phase dextrose in quantity not substantially less than ten parts of the solid to one hundred parts liquid content of the batch.

6. Improvement in the method of making dextrose from a starch converted dextrose solution which consists in inducing crystallization, maintaining the solid phase in substantially uniform dispersion through the solution, and reducing the temperature of the magma when the quantity of the solid phase has increased so as to be a significant factor in controlling crystallization.

7. Improvement in the method of making dextrose from a starch converted dextrose solution which consists in seeding the solution and inducing crystallization, maintaining the solid phase consisting of seed and induced crystals in substantially uniform dispersion through the solution, and reducing the temperature of the magma when the quantity of the solid phase has increased so as to be a significant factor in controlling crystallization.

8. Improvement in the method of making dextrose from a starch converted dextrose solution which consists in seeding the solution and inducing crystallization, maintaining the solid phase in uniform dispersion through the solution and reducing the temperature of the magma when the quantity of the solid phase has increased so as to be a significant factor in controlling crystallization.

9. Improvement in the method of making dextrose from a starch converted dextrose solution which consists in introducing into the solution a sufficient amount of the solid phase dextrose so that the quantity of the solid phase is a significant factor in controlling crystallization.

10. Improvement in the method of making dextrose from a starch converted dextrose solution which consists in introducing into the solution, as seed, a sufficient amount of the solid phase dextrose so that the quantity of the solid phase is a significant factor in controlling crystallization, and maintaining the solid phase dextrose in a state of uniform dispersion throughout the solution.

11. Improvement in the method of making dextrose from a starch converted dextrose solution which consists in introducing into the solution, as seed, a mixture of crystals and mother liquor, in a fluent state, taken from a previous crystallizing operation, in amount sufficient so that the quantity of the solid phase produced is a significant factor in controlling crystallization of the dextrose in solution.

12. Improvement in the method of making dextrose from a starch converted dex-

trose solution having a purity of approximately 89% to 90% which consists in introducing into the solution, as seed, a mixture of crystals and mother liquor, in a fluent state, taken from a previous crystallizing operation, in amount not substantially less than twenty per cent of the batch.

13. Improvement in the method of making dextrose from a starch converted dextrose solution which consists in crystallizing in successive batches and using as seed for each batch (after the first) a portion of the crystallized magma, crystals and mother liquor, produced by the preceding crystallizing operation in such amount that the quantity of the solid phase thus introduced into each batch is a significant factor in controlling crystallization.

14. Improvement in the method of making dextrose from a starch converted dextrose solution which consists in crystallizing in successive batches and using as seed for each batch (after the first) a portion of the crystallized magma, crystals and mother liquor, produced by the preceding crystallizing operation, and mixing the same while in a fluent state with the fresh solution in each case, in such amount that the quantity of the solid phase thus introduced into each batch is a significant factor in controlling the crystallization thereof.

15. Improvement in the method of making dextrose from a starch converted dextrose solution which consists in crystallizing in successive batches and using as seed for each batch (after the first) a portion of the crystallized magma, crystals and mother liquor, produced by the preceding crystallizing operation and mixing the same while in a fluent state with the fresh solution in each case, in such amount that the quantity of the solid phase thus introduced into each batch is a significant factor in controlling the crystallization thereof, and maintaining the solid phase in a state of uniform dispersion throughout the solution from beginning to end of the crystallization.

16. Improvement in the method of making dextrose from starch converted dextrose solution which consists in concentrating the solution by heating, cooling the solution to produce supersaturation, but without appreciable induction of crystals, and then mixing the cooled solution with a quantity of solid phase dextrose.

17. Improvement in the method of making dextrose from a starch converted dextrose solution which consists in concentrating the solution by heating, cooling the solution to produce supersaturation, but without appreciable induction of crystals, and then mixing the cooled solution with a quantity of solid phase dextrose sufficiently large in amount so that its quantity is a significant factor in controlling crystallization.

18. Improvement in the method of making dextrose from a starch converted dextrose solution having a purity of approximately 89% to 90% which consists in concentrating the solution by heating, cooling the solution to produce supersaturation, but without appreciable induction of crystals, and then mixing the cooled solution with a quantity of solid phase dextrose in amount not substantially less than ten parts of the solid to one hundred parts of the liquid in the batch.

19. Improvement in the method of making dextrose from a starch converted dextrose solution which consists in concentrating the solution by heating, cooling the solution to produce supersaturation but without appreciable induction of crystals and then mixing the cooled solution with foots, in a fluent state, taken from a previously crystallized dextrose magma.

20. Improvement in the method of making dextrose from a starch converted dextrose solution which consists in concentrating the solution by heating, cooling the solution to produce supersaturation but without appreciable induction of crystals, and then mixing the cooled solution with foots, in a fluent state, taken from a previously crystallized dextrose magma, in an amount sufficient so that the quantity of the solid phase is a significant factor in controlling crystallization.

21. Improvement in the method of making dextrose from a starch converted dextrose solution having a purity of approximately 89% to 90% which consists in concentrating the solution by heating, cooling the solution to produce supersaturation but without appreciable induction of crystals, and then mixing the cooled solution with foots in a fluent state, taken from a previously crystallized dextrose magma, in an amount not substantially less than twenty per cent of the batch.

22. Improvement in the method of making dextrose from a starch converted dextrose solution which consists in crystallizing in successive batches, heating the solution in each case, to concentrate the same, cooling the concentrated liquor without appreciable induction of crystals and mixing the same with a portion of the crystallized magma of the preceding batch to further reduce the temperature to the proper degree for initiating crystallization.

23. Method of making dextrose from a starch converted dextrose solution which consists in crystallizing in successive batches, heating the solution in each case to concentrate the same, mixing with the concentrated solution foots from a previous batch which are at a lower temperature than that of the solution, and further cooling the magma during the crystallizing period.

24. Method of making dextrose from a starch converted solution which consists in crystallizing in successive batches, heating the solution in each case to concentrate the same, cooling the solution to produce supersaturation without substantial induction of crystals, initiating crystallization by introducing into the solution a body of foots from a previous crystallizing operation at a temperature below the temperature of the solution, and further cooling the magma during the crystallizing period.

25. Improvement in the method of making dextrose hydrate, which consists in introducing into the solution an amount of the solid phase in inverse proportion, substantially, to the quantity of impurities in the solution.

26. Method of making dextrose from a starch converted dextrose solution which comprises concentrating the solution and inducing crystallization in the presence of relatively large quantities of the solid phase, centrifuging to extract mother liquor from the crystals, and inducing crystallization in the extracted mother liquor in the presence of solid phase of smaller amount in proportion to the quantity of the solution treated than was employed in the first mentioned crystallizing operation.

27. Method of making dextrose from a starch converted dextrose solution which comprises concentrating the solution and inducing crystallization in the presence of the solid phase in an amount not substantially less than ten parts to one hundred of the solution used, centrifuging to extract mother liquor from the crystals and inducing crystallization in the extracted mother liquor in the presence of solid phase in an amount not substantially less than eight parts to one hundred of the mother liquor used.

28. Improvement in the method of making dextrose from a starch converted dextrose solution which consists in inducing crystallization in the solution in the presence of dispersed solid phase dextrose in quantity proportioned substantially to the degree of supersaturation maintained and the natural velocity of crystallization of the solution.

29. Method of making dextrose hydrate from a starch converted dextrose solution having a purity of approximately 89% to 90% which comprises concentrating the solution by heat, cooling the same to a temperature within the hydrate range, introducing into the solution foots taken from a previous crystallizing operation in an amount not substantially less than twenty per cent of the new batch, maintaining the solid phase in uniform dispersion throughout the solution, and centrifuging the crystallized magma to extract mother liquor.

30. Method of making dextrose hydrate

from a starch converted dextrose solution having a purity of approximately 89% to 90% which comprises concentrating the solution by heat, cooling the same to a temperature within the hydrate range, introducing into the solution foots taken from a previous crystallizing operation in an amount not substantially less than twenty per cent of the new batch, maintaining the solid phase in uniform dispersion throughout the solution, and reducing the temperature during the crystallization.

31. Improvement in the method of making dextrose from a starch converted solution which consists in introducing into the solution a sufficient amount of solid phase dextrose so that the quantity of the solid phase is a significant factor in controlling crystallization and cooling the mixture to maintain continued supersaturation.

32. Improvement in the method of making dextrose from a starch converted solu-

tion which consists in introducing into the solution a sufficient amount of solid phase dextrose so that the quantity of the solid phase is a significant factor in controlling crystallization and cooling the mixture to maintain continued supersaturation, and maintaining the solid phase in substantially uniform dispersion throughout the solution.

33. Improvement in the method of making dextrose from a starch converted solution which consists in introducing into the solution a sufficient amount of solid phase dextrose so that the quantity of the solid phase is a significant factor in controlling crystallization, cooling the mixture to maintain continued supersaturation, maintaining the solid phase in substantially uniform dispersion throughout the solution, and centrifuging the magma while in a fluent state to separate mother liquor from the crystals.

WILLIAM B. NEWKIRK.